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**(54) Method for bonding semiconductor substrates**

Verfahren zum Bonden von Halbleitersubstraten

Procédé pour la liaison de substrats semi-conducteurs

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## Description

### Field of the Invention

[0001] The present invention is related to the field of semiconductor processing, in particular to methods of bonding semiconductor substrates by a permanent metal bond, for example a permanent Cu/Sn bond. The invention is particularly suitable in the field of LED devices, such as GaNLEDs.

### State of the art.

[0002] One way of producing GaNLED devices is by a transfer process applied after the fabrication of LEDs on 4 inch Si (111) wafers comprising p and n contact formation to the GaN layer. Such a process is described in 'Substrate Transfer for GaN based LEDs grown in Silicon', Pham et al, Proceedings 44th International Symposium on Microelectronics IMAPS 2011, Long Beach CA. After applying a passivation layer, a bonding metal, preferably a Cu/Sn layer is deposited on the GaN layer and on a Si carrier wafer. The wafer is then bonded to said Si carrier wafer using metallic bonding at a temperature of about 250°C. Next, the original Si device wafer is completely removed by grinding and wet etching. GaNLEDs are thus transferred to a new carrier wafer. A typical problem in this type of process is the occurrence of high stress in the Cu layer during bonding and consequently large wafer warpage when the device substrate is removed. Such a large wafer warpage causes problems for some automatic handling tools and processes like lithography. This problem occurs not only in the above-described GaNLED production process, but in any substrate transfer process involving a permanent metal bond such as a Cu/Sn bond between a carrier substrate and a device produced on a temporary device substrate. Document US2005-048739 is related to methods for producing a transfer layer of a semiconductor material on a final substrate. In some embodiments, the transfer layer is produced on the final substrate by forming a layer of semiconductor material on an initial support, assembling that layer and a final substrate by metal bonding, and mechanically separating the initial support from the layer at a weak interface that is initially attached the layer to the initial support. An intermediate substrate can be obtained which can be used to fabricate a variety of components such as light-emitting diodes or laser diodes. Document US2008-0157107 is related to a process for fabricating a light-emitting diode (LED), wherein a bonding process is described involving a metal bonding layer (with reference to paragraph [0022] and Figure 3), formed on the surface of a conducting substrate acting as the carrier substrate onto which a LED device substrate is to be bonded. Ohmic contact layers are described as being selectively formed on two opposite sides of the conductive substrate, to further enhance the electrical quality of the LED device. As examples of the material of the ohmic

contact layer on the back side of the conductive substrate, 'Ti, Ni, Au or W' are given. Possible materials of the metal bonding layer are cited as 'PbSn, AuGe, AuBe, AuSn, Sn, In or PdIn'.

### Summary of the invention

[0003] The invention is related to a method as disclosed in the appended claims.

The invention is thus related to a method for bonding a first substrate having a front and back surface, and carrying a semiconductor device layer on its front surface to a second substrate having a front and back surface, said method comprising the steps of :

- Producing said semiconductor device layer on said front surface of said first substrate,
- Depositing a first metal bonding layer or a stack of metal layers on said first substrate, on top of said semiconductor device layer,
- Depositing a second metal bonding layer or a stack of metal layers on the front surface of said second substrate,
- Establishing a metal bond between said first and second substrate, by bringing said first and second metal bonding layers or stacks of layers into mutual contact under conditions of mechanical pressure and temperature suitable for obtaining said metal bond,
- Removing said first substrate,

wherein a metal stress-compensation layer is deposited on the back side of said second substrate, before the step of establishing a metal bond between said first and second substrate.

A device layer is defined in the context of this description as an essentially continuous layer comprising at least one semiconductor device.

[0004] According to the invention, said stress compensation layer is a single layer of the same material as the metal bonding layer deposited on the front side of the second substrate or of the first layer of said stack of layers.

[0005] The thickness of the stress compensation layer is preferably at least equal to the thickness of the metal bonding layer at the front side of the second substrate, or of the first layer of a stack of layers deposited at said front side of the second substrate.

[0006] According to an embodiment, the thickness of the stress compensation layer is equal to the sum of the thicknesses of the metal bonding layers or of the first layers of layer stacks deposited on the surfaces to be bonded.

[0007] Preferably, said stress compensation layer is deposited on the back side of the second substrate before the step of depositing a second metal bonding layer or a stack of layers on the front side of said second substrate.

[0008] According to an embodiment, a stack of metal bonding layers is deposited on at least one of the surfaces

of the first and second substrate that are to be bonded, and said stack comprises a Cu layer and a Sn layer on top of and in direct contact with the Cu layer.

**[0009]** A protection layer may be deposited on top of said stress-compensation layer. Said protection layer may be a SiN layer.

**[0010]** A diffusion-inhibiting layer may be produced between the second substrate and the stress compensation layer. Said diffusion-inhibiting layer may be a SiN layer.

### **Brief description of the figures**

#### **[0011]**

Figure 1 illustrates a prior art process for transferring a GaNLED device from a first substrate to a second (carrier) substrate.

Figure 2 shows details of the metal bonding steps taking place in the process of figure 1.

Figure 3 illustrates the method according to the invention.

### **Detailed description of the invention**

**[0012]** The invention is related to a process wherein a metal bonding step is applied to establish a bond between a first substrate carrying a semiconductor device, e.g. a GaNLED device, and a second substrate. Hereafter, the first substrate will be referred to as the 'device wafer', and the second substrate as the 'carrier wafer', as the method is firstly applicable to semiconductor wafers. It is emphasized though that the term 'substrate' can be any object that is suitable for being used in a bonding process. In the method of the invention, a metal bonding layer or a stack of metal bonding layers, for example a layer of Cu with a layer of Sn directly on the Cu layer is deposited on each of the surfaces that are to be bonded. According to the invention, a stress compensation layer is provided at the backside of the carrier wafer, before the metal bonding step is performed. Preferably, the stress compensation layer is a single layer of the same material as the metal bonding layer deposited on the front side (i.e. the side to be bonded), of the carrier wafer. If a stack of bonding layers is deposited on said front side, the material of the compensation layer is the same as that of the first layer of said stack ('first' being at the bottom of the stack). The thickness of the stress compensation layer is at least equal to the thickness of the metal layer at the front side of the carrier wafer, or of the first layer of a stack of layers deposited at said front side of the carrier. According to a more preferred embodiment, the thickness of the stress compensation layer is equal to the sum of the thicknesses of the metal layers or of the first layers of layer stacks deposited on the surfaces to be bonded (i.e. the surfaces of the device wafer and the carrier wafer).

**[0013]** For example, when a Cu/Sn stack is deposited on the device wafer and on the carrier wafer, the stress

compensation layer is a Cu layer the thickness of which is the sum of the Cu layers on the device and the carrier wafers. If one wafer comprises a single bonding layer and the other a stack of bonding layers, the latter embodiment signifies that the thickness of the stress compensation layer is equal to the sum of the thicknesses of the single bonding layer and the first of the stack of layers.

**[0014]** In the method of the invention, the stress compensation layer at the back of the carrier wafer undergoes the same changes in stress as the bonding layer of the same material at the front of the carrier. Any tensile stress appearing in the bonding layer that would induce warpage is thereby compensated, so that the carrier wafer remains flat throughout the bonding process, and after the removal of the device wafer.

**[0015]** A preferred embodiment is described hereafter in more detail. This is the case described in the above cited reference (Pham et al.), wherein the semiconductor device is a GaNLED device, produced on a first Si-wafer, and bonded to a second (carrier) Si wafer by establishing a Cu/Sn bond. Figure 1 gives an overview of the bonding steps according to this method. All parameter values given hereafter are cited by way of example only and are not limiting the scope of the invention. First, the GaNLED device layer 1 is produced on a Si(111) wafer 2 (Fig. 1a). This may take place by the following steps, as known in the art :

1. epitaxial growth of GaN-based LED layers,
2. dopant activation anneal,
3. dry etching of the LED mesa
4. patterning of Ag-based p-type and Ti/Al-based n-type contacts
5. deposition of a CVD SiO<sub>2</sub> inter-metal dielectric (IMD)
6. patterning of contact holes towards the p- and n-type electrodes using dry etch,
7. deposition and patterning of a Au-based interconnect layer
8. deposition of a CVD SiO<sub>2</sub> passivation layer

**[0016]** The GaN-based LED layers may be deposited using a 4 inch Aixtron MOCVD epi reactor. The total stack thickness is about 3.7 μm and consists of a buffer layer comprising AlN/AlGaIn/GaN layers to reduce defect density, n-type Si-doped and p-type Mg-doped GaN cladding layers, multiple InGaIn quantum wells (MQW) and an InGaIn electron blocking layer (EBL). The mesa etch removes the pGaIn, EBL, MQW and part of the nGaIn layer such that the remaining thickness of the GaIn stack in field regions is 2.8 to 3 μm.

**[0017]** Then the Cu/Sn bonding layers are produced, see figure 1b and illustrated in more detail in figure 2 (in figure 2, the GaIn stack 9 is not shown for the sake of simplicity). A seed layer for Cu deposition (e.g. 30nm TiW/150nm, not shown) may be deposited on top of the oxide passivation of the device wafer. The same type of seed layer may be deposited directly on the Si

(100) carrier wafer. After seed layer deposition, a 5 µm Cu layer 11 followed by a 10 µm Sn layer 12 are deposited on the device wafer using electroplating. The same layer of Cu/Sn is then electroplated on the carrier wafer 20. Other compositions of the metal layers may be used, and other thicknesses of these layers. For example, one surface may receive a Cu/Sn layer while the other receives only a Cu layer. The advantage of using a Cu/Sn layer on both sides is that it does not require any cleaning treatment of the Sn surface before bonding. Also, the Cu layers 11 on the device wafer and the carrier wafer may have different thicknesses, although in general the Cu bonding layer on both device and carrier have the same thickness.

**[0018]** After metallization, the two wafers are bonded (fig. 1c) in a suitable apparatus, e.g. an EVG 520 bonder, using a sequence as shown in Fig 2. First, the two wafers are brought into contact. Then, the temperature of the bonding chuck is raised to 250°C and maintained for 10min. At the same time, a load of 1000N is applied to the wafer stack. Again, these parameters are mere examples and not limiting to the invention scope. The metallic bonding is made by diffusion soldering (or transient liquid phase soldering) of the Cu-Sn system. The solder layers 12 (Sn) melt and diffuse into the two parent layers (Cu) at moderate temperatures (250°C), thereby forming a layer 13, comprising a central layer 13' of copper-tin intermetallics ( $\text{Cu}_x\text{Sn}_y$ ), and still a layer 11' of Cu on either side (the thickness of the Cu layers 11' being normally smaller than the thickness of the original Cu layers 11). The  $\text{Cu}_x\text{Sn}_y$  intermetallics have a much higher melting temperature (>415°C) than the original Sn (232°C). This provides an advantage such that, during any subsequent (high temperature) processing, the metallic seal remains in a solid state.

**[0019]** Then the Si(111) device wafer 2 is removed in two processing steps: Thinning down the Si device wafer to a thickness of ~ 100 µm or lower (Fig 1d) and wet etching of the remaining Si (Fig 1e). Thinning of the Si wafer from the original thickness of 1000 µm down to 100 µm may be performed by grinding or lapping and polishing. To further remove the Si device wafer, an isotropic Si wet etching process may be applied using an HNA solution (etch solution composed of HF: HNO<sub>3</sub>: CH<sub>3</sub>COOH). These steps are then followed by a texturization step (not shown), and by the opening of the contacts 21 (fig. 1f).

**[0020]** The method of the invention as applied to the GaNLED fabrication process of figure 1 is illustrated in figure 3. A stress compensation layer 30 is deposited (e.g. by electroplating) on the backside of the carrier wafer 20. The stress compensation layer 30 is a Cu layer, the thickness of which is essentially the sum of the Cu layers 11 on the device wafer 2 and on the carrier wafer 20.

**[0021]** Preferably and as shown in the embodiment of figure 3, the stress compensation layer 30 is deposited before the bonding layers 11/12 are deposited at the front of the carrier wafer 20. Alternatively, the compensation

layer 30 may be deposited after the bonding layers 11/12.

**[0022]** Also in the embodiment shown, a further layer 31 is deposited on the compensation layer. This further layer is a protection layer configured to prevent direct contact between the production tools and the Cu compensation layer 30 during the further processing and in this way to prevent spreading Cu contamination. The latter is important especially at silicon processing production facilities. This protection layer may be a SiN layer, deposited by PECVD (Plasma Enhanced Chemical Vapour Deposition).

**[0023]** The actual bonding process takes place in the same way as in the prior art method described above. The device wafer 2 is bonded to the carrier wafer 20 and then removed by thinning and wet etching. After bonding, and after grinding and etching the Si device wafer 2, no warping of the carrier 20 takes place due to the presence of the stress compensation layer 30.

**[0024]** Another embodiment is relevant for the GaNLED application described above, and also for CMOS applications. According to the latter embodiment, an intermediate diffusion-inhibiting layer is produced between the backside of the carrier wafer 20 and the stress compensation layer 30. When the stress compensation layer is a Cu layer for example, the intermediate layer may be a SiN layer produced by PECVD on the back side of the carrier wafer, the stress compensation layer being deposited on the SiN layer. The SiN layer is configured to inhibit diffusion of Cu from the stress compensation layer into the carrier wafer. According to a specific embodiment, an intermediate diffusion-inhibiting layer as described above is present on one side of the stress compensation layer 30, and a protection layer 31 is present on the other. Both the diffusion-inhibiting layer and the protection layer may be SiN layers.

**[0025]** The carrier wafer 20 as obtained in the last step of figure 3 may be further processed and eventually divided into individual devices, e.g. individual LEDs. The invention is thus related to such devices, comprising a device portion and a wafer portion and which are characterized by the presence of a stress compensation layer on the back of the wafer portion. Possibly, a protection layer and/or an anti-diffusion layer may be present on top of or underneath the stress compensation layer respectively.

**[0026]** While the invention has been illustrated and described in detail in the drawings and foregoing description, such illustration and description are to be considered illustrative or exemplary and not restrictive. Other variations to the disclosed embodiments can be understood and effected by those skilled in the art in practicing the claimed invention, from a study of the drawings, the disclosure and the appended claims. In the claims, the word "comprising" does not exclude other elements or steps, and the indefinite article "a" or "an" does not exclude a plurality. The mere fact that certain measures are recited in mutually different dependent claims does not indicate that a combination of these measures cannot be used to

advantage. Any reference signs in the claims should not be construed as limiting the scope. Unless specifically specified, the description of a layer being deposited or produced 'on' another layer or substrate, includes the options of

- said layer being produced or deposited directly on, i.e. in contact with, said other layer or substrate, and
- said layer being produced on one or a stack of intermediate layers between said layer and said other layer or substrate.

**[0027]** The foregoing description details certain embodiments of the invention. It will be appreciated, however, that no matter how detailed the foregoing appears in text, the invention may be practiced in many ways, and is therefore not limited to the embodiments disclosed. It should be noted that the use of particular terminology when describing certain features or aspects of the invention should not be taken to imply that the terminology is being re-defined herein to be restricted to include any specific characteristics of the features or aspects of the invention with which that terminology is associated.

## Claims

1. A method for bonding a first substrate (2) having a front and back surface, and carrying a semiconductor device layer (1) on its front surface to a second substrate (20) having a front and back surface, said method comprising the steps of :

- Producing said semiconductor device layer (1) on said front surface of said first substrate (2),
- Depositing a first metal bonding layer or a stack of metal layers (11,12) on said first substrate, on top of said semiconductor device layer,
- Depositing a second metal bonding layer or a stack of metal layers (11,12) on the front surface of said second substrate (20),
- Establishing a metal bond between said first and second substrate, by bringing said first and second metal bonding layers or stacks of layers into mutual contact under conditions of mechanical pressure and temperature suitable for obtaining said metal bond,
- Removing said first substrate (2),

**characterized in that** a metal stress-compensation layer (30) is deposited on the back side of said second substrate (20), before the step of establishing a metal bond between said first and second substrate (2, 20), wherein said stress compensation layer (30) is a single layer of the same material as the metal bonding layer deposited on the front side of the second substrate or of the first layer of said stack of

layers.

2. Method according to claim 1, wherein the thickness of the stress compensation layer (30) is at least equal to the thickness of the metal bonding layer at the front side of the second substrate, or of the first layer (11) of a stack of layers deposited at said front side of the second substrate.
3. Method according to claim 1 or 2, wherein the thickness of the stress compensation layer (30) is equal to the sum of the thicknesses of the metal bonding layers or of the first layers (11) of layer stacks deposited on the surfaces to be bonded.
4. Method according to any one of claims 1 to 3, wherein said stress compensation layer is deposited on the back side of the second substrate before the step of depositing a second metal bonding layer or a stack of layers on the front side of said second substrate.
5. Method according to any one of claims 1 to 4, wherein a stack of metal bonding layers (11,12) is deposited on at least one of the surfaces of the first and second substrate (2,20) that are to be bonded, and wherein said stack comprises a Cu layer (11) and a Sn layer (12) on top of and in direct contact with the Cu layer.
6. Method according to any one of claims 1 to 5, wherein a protection layer (31) is deposited on top of said stress-compensation layer (30).
7. Method according to claim 6, wherein said protection layer (31) is a SiN layer.
8. Method according to any one of claims 1 to 7, wherein a diffusion-inhibiting layer is arranged between the second substrate (20) and the stress compensation layer (30) .
9. Method according to claim 8, wherein said diffusion-inhibiting layer is a SiN layer.
10. Method according to any one of claims 1 to 8, wherein said semiconductor device layer comprises at least one GaN LED device.

## Patentansprüche

1. Verfahren zum Bondieren eines ersten Substrats (2), das eine vordere und hintere Oberfläche aufweist und eine Halbleitervorrichtungsschicht (1) auf seiner vorderen Oberfläche trägt, an ein zweites Substrat (20), das eine vordere und hintere Oberfläche aufweist, wobei das Verfahren die Schritte umfasst des:

- Herstellens der Halbleitervorrichtungsschicht (1) auf der vorderen Oberfläche des ersten Substrats (2),
- Absetzens einer ersten Metallbondierschicht oder eines Stapels von Metallschichten (11, 12) auf dem ersten Substrat über der Halbleitervorrichtungsschicht,
- Absetzens einer zweiten Metallbondierschicht oder eines Stapels von Metallschichten (11, 12) auf der vorderen Oberfläche des zweiten Substrats (20),
- Bildens einer Metallbindung zwischen dem ersten und zweiten Substrat durch gegenseitiges Inkontaktbringen der ersten und zweiten Metallbondierschichten oder Stapel von Schichten unter Bedingungen von mechanischem Druck und Temperatur, die für das Erhalten der Metallbindung geeignet sind,
- Entfermens des ersten Substrats (2),

**dadurch gekennzeichnet, dass**

eine Metallspannungskompensationsschicht (30) auf der Rückseite des zweiten Substrats (20) vor dem Schritt des Bildens einer Metallbindung zwischen dem ersten und zweiten Substrat (2, 20) abgesetzt wird, wobei die Spannungskompensationsschicht (30) eine einzelne Schicht desselben Materials wie die Metallbondierschicht ist, die auf der Vorderseite des zweiten Substrats oder der ersten Schicht des Stapels von Schichten abgesetzt ist.

2. Verfahren nach Anspruch 1, wobei die Dicke der Spannungskompensationsschicht (30) mindestens gleich der Dicke der Metallbondierschicht auf der Vorderseite des zweiten Substrats oder der ersten Schicht (11) eines Stapels von Schichten, der auf der Vorderseite des zweiten Substrats abgesetzt ist, ist.
3. Verfahren nach Anspruch 1 oder 2, wobei die Dicke der Spannungskompensationsschicht (30) gleich der Summe von Dicken der Metallbondierschichten oder der ersten Schichten (11) von Schichtstapeln, die auf den zu bondierenden Oberflächen abgesetzt sind, ist.
4. Verfahren nach einem der Ansprüche 1 bis 3, wobei die Spannungskompensationsschicht auf der Rückseite des zweiten Substrats vor dem Schritt des Absetzens einer zweiten Metallbondierschicht oder eines Stapels von Schichten auf der Vorderseite des zweiten Substrats abgesetzt wird.
5. Verfahren nach einem der Ansprüche 1 bis 4, wobei ein Stapel von Metallbondierschichten (11, 12) auf mindestens einer der Oberflächen des ersten und zweiten Substrats (2, 20), die bondiert werden sol-

len, abgesetzt wird und wobei der Stapel eine Cu-Schicht (11) und eine Sn-Schicht (12) über und in direktem Kontakt mit der Cu-Schicht umfasst.

6. Verfahren nach einem der Ansprüche 1 bis 5, wobei eine Schutzschicht (31) über der Spannungskompensationsschicht (30) abgesetzt wird.
7. Verfahren nach Anspruch 6, wobei die Schutzschicht (31) eine SiN-Schicht ist.
8. Verfahren nach einem der Ansprüche 1 bis 7, wobei eine diffusionshemmende Schicht zwischen dem zweiten Substrat (20) und der Spannungskompensationsschicht (30) angeordnet ist.
9. Verfahren nach Anspruch 8, wobei die diffusionshemmende Schicht eine SiN-Schicht ist.
10. Verfahren nach einem der Ansprüche 1 bis 8, wobei die Halbleitervorrichtungsschicht mindestens eine GaN-LED-Vorrichtung umfasst.

**Revendications**

1. Procédé pour lier un premier substrat (2) ayant des surfaces avant et arrière, et portant une couche de dispositif semi-conducteur (1) sur sa surface avant à un second substrat (20) ayant des surfaces avant et arrière, ledit procédé comprenant les étapes de :

- production de ladite couche de dispositif semi-conducteur (1) sur ladite surface avant dudit premier substrat (2),
- dépôt d'une première couche de liaison métallique ou d'un empilement de couches métalliques (11, 12) sur ledit premier substrat, au-dessus de ladite couche de dispositif semi-conducteur,
- dépôt d'une seconde couche de liaison métallique ou d'un empilement de couches métalliques (11, 12) sur la surface avant dudit second substrat (20),
- établissement d'une liaison métallique entre lesdits premier et second substrats, en mettant en contact mutuel lesdits première et seconde couches de liaison métallique ou empilements de couches dans des conditions de pression mécanique et de température appropriées pour obtenir ladite liaison métallique,
- retrait dudit premier substrat (2),

**caractérisé en ce qu'**une couche de compensation de contrainte métallique (30) est déposée sur le côté arrière dudit second substrat (20), avant l'étape d'établissement d'une liaison métallique entre lesdits premier et second substrats (2, 20), dans lequel

ladite couche de compensation de contrainte (30) est une couche unique du même matériau que la couche de liaison métallique déposée sur le côté avant du second substrat ou de la première couche dudit empilement de couches.

5

2. Procédé selon la revendication 1, dans lequel l'épaisseur de la couche de compensation de contrainte (30) est au moins égale à l'épaisseur de la couche de liaison métallique au niveau du côté avant du second substrat, ou de la première couche (11) d'un empilement de couches déposées au niveau dudit côté avant du second substrat. 10
3. Procédé selon la revendication 1 ou 2, dans lequel l'épaisseur de la couche de compensation de contrainte (30) est égale à la somme des épaisseurs des couches de liaison métalliques ou des premières couches (11) d'empilements de couches déposées sur les surfaces à lier. 15 20
4. Procédé selon l'une quelconque des revendications 1 à 3, dans lequel ladite couche de compensation de contrainte est déposée sur le côté arrière du second substrat avant l'étape de dépôt d'une seconde couche de liaison métallique ou d'un empilement de couches sur le côté avant dudit second substrat. 25
5. Procédé selon l'une quelconque des revendications 1 à 4, dans lequel un empilement de couches de liaison métalliques (11, 12) est déposé sur au moins l'une des surfaces des premier et second substrats (2, 20) qui doivent être liés, et dans lequel ledit empilement comprend une couche de Cu (11) et une couche de Sn (12) au-dessus de et en contact direct avec la couche de Cu. 30 35
6. Procédé selon l'une quelconque des revendications 1 à 5, dans lequel une couche de protection (31) est déposée au-dessus de ladite couche de compensation de contrainte (30). 40
7. Procédé selon la revendication 6, dans lequel ladite couche de protection (31) est une couche de SiN. 45
8. Procédé selon l'une quelconque des revendications 1 à 7, dans lequel une couche inhibitrice de diffusion est agencée entre le second substrat (20) et la couche de compensation de contrainte (30). 50
9. Procédé selon la revendication 8, dans lequel ladite couche inhibitrice de diffusion est une couche de SiN.
10. Procédé selon l'une quelconque des revendications 1 à 8, dans lequel ladite couche de dispositif semi-conducteur comprend au moins un dispositif à LED au GaN. 55

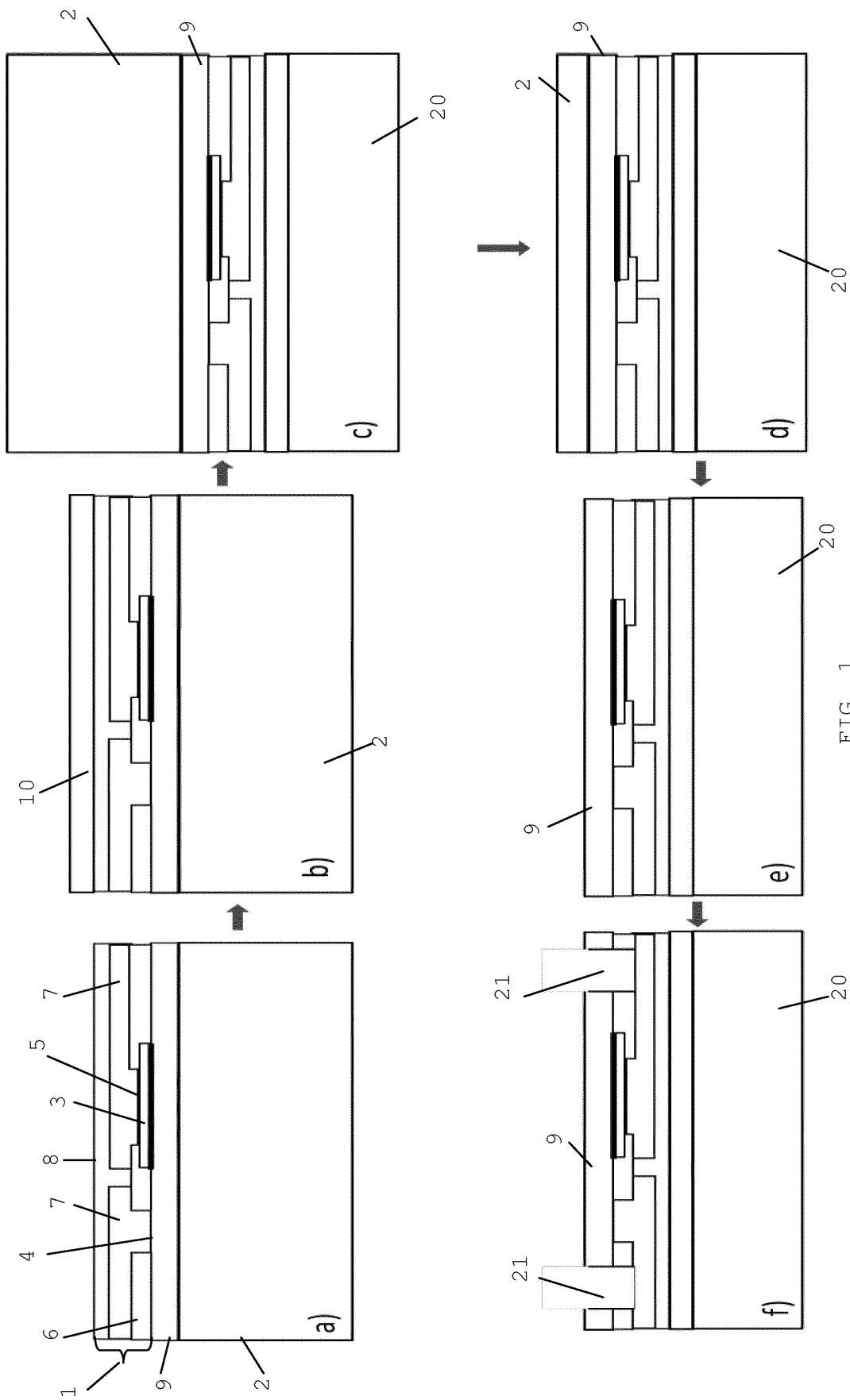


FIG. 1



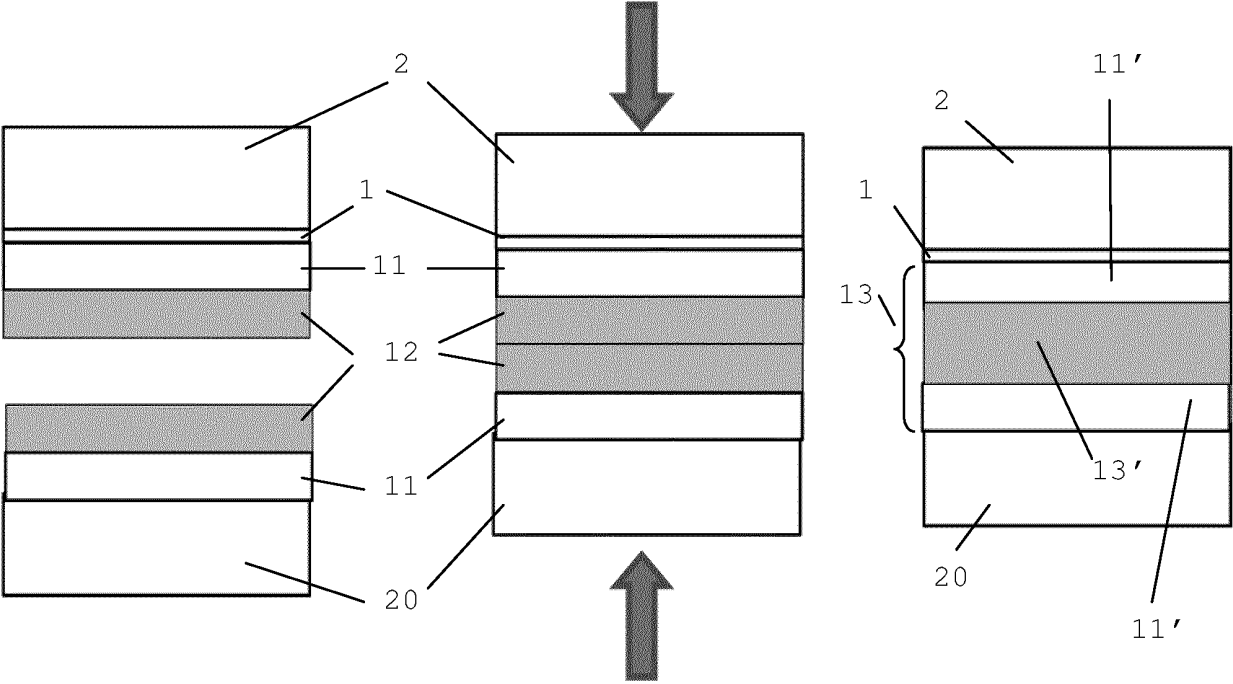


FIG. 2

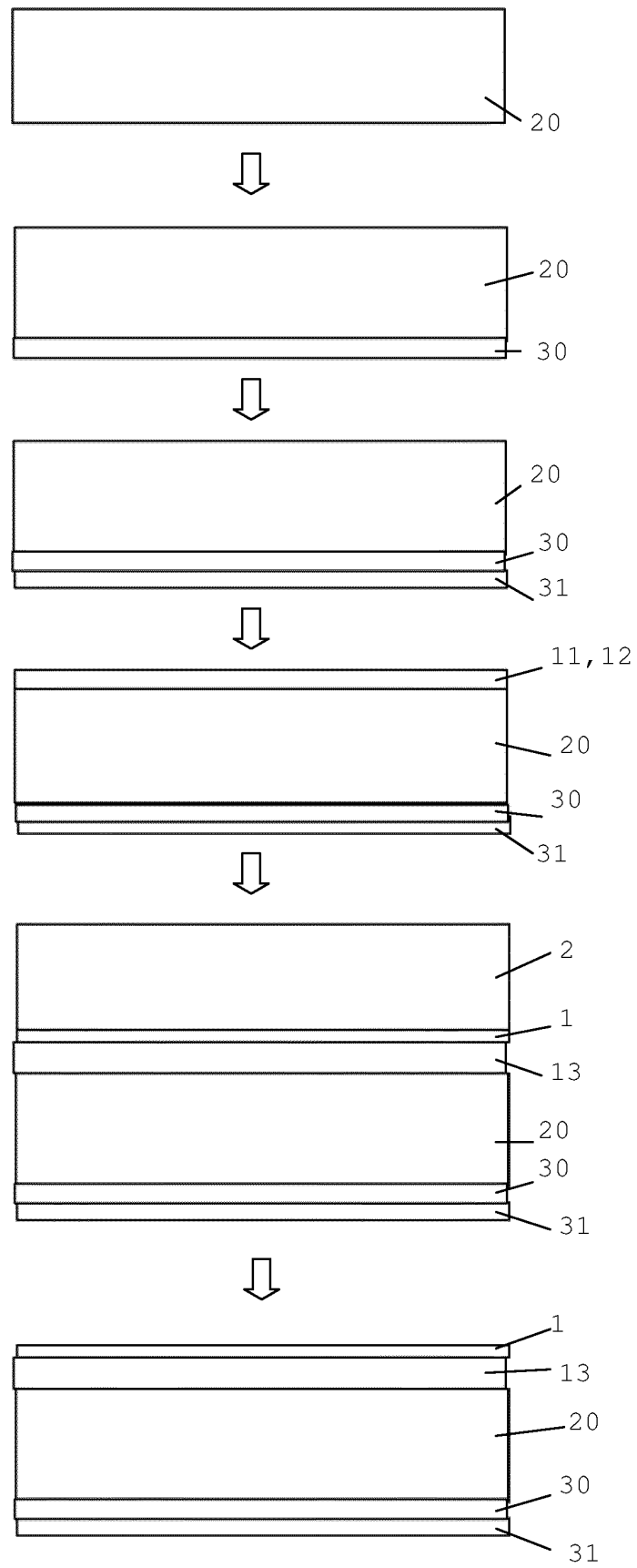


FIG. 3

**REFERENCES CITED IN THE DESCRIPTION**

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